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In accordance to our telephone conversation, please find enclosed the last three pages of the Declaration Under 37 CFR 1.132 (Dr. Alfred KAIZIK).

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O.Z. 6219-W0/US
Additional comparative example

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Co-
Example 4 (variant B/~~catalyst~~catalyst)

Di-n-butene (560 g) was hydroformylated in the presence of a cobalt catalyst using a method analogous to example 1 of the original patent application. Cobalt(II) 2-ethylhexanoate was used as catalyst precursor. Apart from the olefin, 560 g of propylene carbonate and 1.505 g or 3.01 g of cobalt(II) 2-ethylhexanoate (65% strength solution in 2-ethylhexanol), corresponding to a cobalt concentration of 150 ppm or 300 ppm of cobalt based on the mass of the reactor contents, were placed in the reactor. The experiments were carried out at a reaction pressure of 250 bar and reaction temperatures of 160, 170 and 180°C. Unlike the experiments using an Rh catalyst (example 2), the reactor outputs of experiments 16 to 21 had two phases. All reactor outputs were slightly yellowish. Propylene carbonate and cobalt were detected in both phases. The composition of the lighter hydrocarbon phase was determined by gas chromatography after work-up, as in example 1. The results of the gas chromatography and the reaction conditions such as temperature, pressure and cobalt concentration are summarized in table 4.

Table 4: Hydroformylation of di-n-butene at 250 bar and various temperatures for 5 hours. The proportions (in % by mass) reported relate to the composition of the lighter hydrocarbon phase, excluding any carbonic esters present.

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No.	T/°C	c(Co)/ ppm	C8-hydro-carbon/ %	C9-alde-hyde/ %	C9-alco-hol/ %	For-mates/ %	High boilers/ %
16	160	150	11.5	79.0	3.2	2.1	4.2
17	170	150	9.7	75.1	5.6	3.8	5.8

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 Additional comparative example

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18	180	150	8.6	71.5	7.2	5.2	7.5
19	160	300	9.6	68.4	10.7	3.8	7.5
20	170	300	8.4	57.1	18.8	7.2	8.5
21	180	300	8.1	43.5	31.7	7.5	9.2

To obtain an approximately equal activity of the catalyst, significantly higher concentrations of metal had to be employed when using the cobalt catalyst in example 4 than when using the rhodium catalyst in example 2. In example 4, the two phases in each of which propylene carbonate and cobalt are present are presumably formed because of the reduced formation of C₉-alcohols when using the cobalt catalyst. The separation of the cobalt from the two phases is more complicated than the work-up of the single-phase reaction mixture from example 2. Although two phases are obtained in example 1, the rhodium is mostly present in the propylene carbonate phase which can be directly recirculated.

As can be seen from table 4, the di-n-butene is hydroformylated significantly more slowly when using cobalt as catalyst compared to the hydroformylation using the rhodium catalyst (example 2, table 2) at 170°C and a comparable reaction pressure and the selectivity is also noticeably poorer. After a time of 5 hours, the C₈-hydrocarbons are present in a concentration of 8.4 or 9.7% by mass, depending on the cobalt concentration used, which is significantly higher than in the hydroformylation (experiments 10 and 11) using the Rh catalyst, in which case C₈-hydrocarbon concentrations of 4.0 or 4.5% by mass were obtained. The selectivity of the hydroformylation of di-n-butene using a Co catalyst is reduced by formation of esters (isononyl formates) and high boilers.

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Additional comparative example

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It could thus be shown that the hydroformylation in the presence of propylene carbonate leads to significantly better results when using the rhodium catalyst than when using the cobalt catalyst.